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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification ⁶ : A61L 2/20, B29C 71/00, D04H 13/00</p>	<p>A1</p>	<p>(11) International Publication Number: WO 96/00093 (43) International Publication Date: 4 January 1996 (04.01.96)</p>
<p>(21) International Application Number: PCT/US95/07716 (22) International Filing Date: 26 June 1995 (26.06.95) (30) Priority Data: 08/266,293 27 June 1994 (27.06.94) US (71) Applicant: KIMBERLY-CLARK CORPORATION [US/US]; 401 North Lake Street, Neenah, WI 54956 (US). (72) Inventor: COHEN, Bernard; 381 Lakeshore Drive, Berkeley Lake, GA 30136 (US). (74) Agents: ALEXANDER, David, J. et al.; Kimberly-Clark Corporation, 401 North Lake Street, Neenah, WI 54956 (US).</p>		<p>(81) Designated States: AM, AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, JP, KE, KG, KP, KR, KZ, LK, LR, LT, LU, LV, MD, MG, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SI, SK, TJ, TT, UA, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG), ARIPO patent (KE, MW, SD, SZ, UG).</p> <p>Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>
<p>(54) Title: IMPROVED NONWOVEN BARRIER AND METHOD OF MAKING THE SAME</p> <p>(57) Abstract</p> <p>An ethylene oxide sterilizable nonwoven material which is subjected to charging, and more particularly, electrostatic charging is provided. The nonwoven materials may include laminate nonwovens wherein one or more layers are subjected to charging. The nonwoven material(s) may also be treated with an antistatic material before or after subjecting the same to charging.</p>		

IMPROVED NONWOVEN BARRIER
AND
METHOD OF MAKING THE SAME

5

FIELD OF THE INVENTION

The present invention is directed to bacterial barrier fabrics. More particularly, the present invention is directed to nonwoven bacterial barrier fabrics for use as, for example, sterilization wrap, surgical draping, surgical gowns, cover garments, such as over-suits, and the like.

BACKGROUND OF THE INVENTION

As is generally known, surgical gowns, surgical drapes, surgical face masks and sterile wrap (hereinafter collectively "surgical articles") have been designed to greatly reduce, if not prevent, the transmission through the surgical article of liquids and/or airborne contaminants. In surgical procedure environments, such liquid sources include the gown wearer's perspiration, patient liquids, such as blood and life support liquids such as plasma and saline. Examples of airborne contaminants include, but are not limited to, biological contaminants, such as bacteria, viruses and fungal spores. Such contaminants may also include particulate material such as, but not limited to, lint, mineral fines, dust, skin squames and respiratory droplets. A measure of a fabrics ability to prevent the passage of such airborne materials is sometimes expressed in terms of "filtration efficiency".

Many of these surgical articles were originally made of cotton or linen and were sterilized prior to their use in the operating room. Such surgical articles fashioned from these materials, however, permitted transmission or "strike-through" of various liquids encountered in surgical procedures. In these instances, a path was established for transmission of biological contaminants, either present in the liquid or subsequently contacting the liquid, through the surgical article. Additionally, in many instances

surgical articles fashioned from cotton or linen provide insufficient barrier protection from the transmission therethrough of airborne contaminants. Furthermore, these articles were costly, and, of course, laundering and sterilization procedures were required before reuse.

Disposable surgical articles have largely replaced linen surgical articles. Advances in such disposable surgical articles include the formation of such articles from totally liquid repellent fabrics which prevent strike-through. In this way, biological contaminants carried by liquids are prevented from passing through such fabrics. However, in some instances, surgical articles formed from nonporous films, while being liquid and airborne contaminate impervious, may retain body heat and moisture and thus may become over a period of time, uncomfortable to wear.

In some instances, surgical articles fashioned from liquid repellent fabrics, such as fabrics formed from nonwoven polymers, sufficiently repel liquids and are more breathable and thus more comfortable to the wearer than nonporous materials. However, these improvements in comfort and breathability provided by such nonwoven fabrics have generally occurred at the expense of barrier properties or filtration efficiency.

While the focus thus far has been directed to surgical articles, there are many other garment or over-garment applications, such as personal protective equipment applications, whose designers require both fabric comfort and filtration efficiency. Other personal protective equipment applications include, but are not limited to, laboratory applications, clean room applications, such as semi-conductor manufacture, agriculture applications, mining applications, and environmental applications.

Therefore, there is a need for garment materials and methods for making the same which provide improved breathability and comfort as well as improved filtration efficiency. Such improved materials and methods are

provided by the present invention and will become more apparent upon further review of the following specification and claims.

5 SUMMARY OF THE INVENTION

 In response to the above problems encountered by those of skill in the art, the present invention provides an ethylene oxide sterilizable polymer web, such as, for example, a nonwoven fabric. The webs of the present invention are formed by subjecting a portion of the web to charging, and more particularly to electrostatic charging, and then ethylene oxide sterilizing the web. The web may be subjected to charging followed by ethylene oxide sterilization or ethylene oxide sterilization followed by charging. The web may also be treated with an antistatic material before or after subjecting the web to charging.

 The above web may further include a second web in a juxtaposed relationship to the first web. The second web may be formed from polymer fibers wherein a portion of these fibers may be subjected to charging. An antistatic treatment may also be present about portions of the second web.

25 DETAILED DESCRIPTION OF THE INVENTION

 Disclosed herein are compositions, and methods of making the same, which improved both the airborne contaminate barrier and filtration efficiency of a web. The web of the present invention may be formed from polymer fibers, films, foams or a combination thereof. The films and foams may be porous or non-porous.

 Among the applications for such compositions and methods are included, but not limited to, applications requiring sterilizable, breathable materials having high airborne contaminate barrier properties. Such materials have application in surgical articles, such as gowns, drapes, sterile wrap and face mask, as well as other non-surgical

applications such as agriculture, mining, clean room and environmental.

5 Polymers, and particularly thermoplastic polymers, are well suited for the formation of webs which are useful in the practice of the present invention. Nonwoven webs useful in present invention can be made from a variety of processes including, but not limited to, air laying processes, wet laid processes, hydroentangling processes, spunbonding, meltblowing, staple fiber carding and bonding, 10 and solution spinning.

 The materials suitable for forming webs of the present invention include a variety of dielectric materials such as, but not limited to, polyesters, polyolefins, nylon and copolymers, polymer blends and bi-component polymers of 15 these materials. In the case of nonwoven webs formed from fibers, the fibers may be relatively short, staple length fibers, typically less than 3 inches, or longer more continuous fibers such as are produced by a spunbonding process.

20 It has been found that nonwoven webs formed from polyolefin-based fibers are particularly well-suited for the above applications. Examples of such nonwovens are the polypropylene nonwovens produced by Kimberly-Clark Corporation. And more particularly, a three layered the 25 spunbond, meltblown, spunbond material (SMS) produced by Kimberly-Clark Corporation.

 This spunbond, meltblown, spunbond material may be made from three separate layers which are laminated to one another. Such a method of making this laminated material is 30 described in commonly assigned U.S. Patent NO. 4,041,203 to Brock et al which is herein incorporated by reference. Alternatively, the spunbond, meltblown, spunbond material may be made by first forming a spunbond, meltblown laminate. The spunbond, meltblown laminate is formed by 35 applying a layer of meltblown on to a layer of spunbond. The second layer of spunbond is then applied to the meltblown side of the previously formed spunbond, meltbl wn

laminate. Generally, the two outer layers provide the nonwoven fabric with strength while the inner layer provides barrier properties.

Suitable webs may be formed from a single layer or multiple layers. In the case of multiple layers, the layers are generally positioned in a juxtaposed or surface-to-surface relationship and all or a portion of the layers may be bound to adjacent layers. In the case of a nonwoven web, the nonwoven web may be formed from a plurality of separate nonwoven webs wherein the separate nonwoven webs may be formed from single or multiple layers. In those instances where the web includes multiple layers, the entire thickness of the web may be subjected to charging or individual layers may be separately subjected to charging and then combined with other layers in a juxtaposed relationship to form the finished web.

There are many well known methods of subjecting a material to charging, and particularly electrostatic charging. These well known methods include, for example, thermal, liquid-contact, electron beam and corona discharge methods. The method used for electrostatically charging the materials discussed in the Examples 1 and 2 (below) is the technique disclosed in U.S. Patent Application No. 07/958,958 filed October 9, 1992 which is assigned to the University of Tennessee, and is herein incorporated by reference. This technique involves subjecting a material to a pair of electrical fields wherein the electrical fields have opposite polarities.

Sterilization of the web may also be accomplished by ethylene oxide sterilization. In those instances when it is desired to sterilize surgical instruments by ethylene oxide, the surgical instruments may be wrapped in a nonwoven web. The entire package may then be subjected to an ethylene oxide sterilization cycle. When the ethylene oxide sterilization cycle is completed, the instruments, still wrapped, are then removed from the ethylene oxide sterilizing equipment and are stored in the wrapping

material until needed. When needed, the wrapping web is removed making the instruments available for handling.

5 The ethylene oxide sterilization cycle may vary dependent upon type of sterilizer and the size/quantity of the items being sterilized. In the Examples described below, ethylene oxide sterilization was accomplished by using either a RSSA Chamber J88-39 or J88-59, made by Vacu Dyne, IL. Generally, the ethylene oxide sterilization
10 cycle includes a preconditioning phase, a sterilization phase and a de-gassing phase. The process parameters for each of these phases are provided below.

15

A. PRECONDITIONING

	<u>Process Parameters</u>	<u>Set Point</u>
20	Temperature	115°F
	Relative Humidity	63%
25	Holding time	18 hours

B. STERILIZATION

5	<u>Process Parameters Set Point</u>	
	Chamber Temperature during exposure	130.0 F
10	Chamber Temperature at all other times	130.0 F
	Initial Evacuation	1.2" Absolute
15	Leak Test	1.2" Absolute
	Leak Test Dwell	5 minutes
	Nitrogen Dilution	3.2" Absolute
20	Evacuation	1.2" Absolute
	Humidity Injection Pressure Increase to	2.9" Absolute
25	Humidification Dwell Time	30 minutes
	ETC Injection Pressure	15" Absolute
30	Time to inject gas	NA
	Cycle Exposure	2 hours
35	Exposure Pressure	15" Absolute
	Exposure Temperature	130.0 F
	1st Re-evacuation	6.0" Absolute
40	1st Nitrogen Inbleed	50.0" Absolute
	2nd Re-evacuation	1.6" Absolute
45	2nd Nitrogen Inbleed	50.0" Absolute
	3rd Re-evacuation	1.6" Absolute
	3rd Nitrogen Inbleed	50.0 Absolute
50	4th Re-evacuation	1.6" Absolute
	Air Inbleed	To Atmospheric Pressure
55		

C. DEGASSING PARAMETERS

<u>Process Parameter</u>	<u>Set Point</u>
Degassing Time	24.0 hours
Degassing Temperature	130° F

10

In those instances where the web is used in or around flammable materials or static charge build-up and/or discharge is a concern, the web may be treated with any number of antistatic materials. In these instances, the antistatic material may be applied to the web by any number of well known techniques including, but not limited to dipping the web into a solution containing the antistatic material or by spraying the web with a solution containing the antistatic material. In some instances the antistatic material may be applied to both the external surfaces of the web and the bulk of the web. In other instances, the antistatic material may be applied to portions of the web, such as a selected surface or surfaces thereof.

Of particular usefulness as an antistatic material is an alcohol phosphate salt product known as ZELEC® and available from the Du Pont Corporation. The web may be treated with the antistatic material either before or after subjecting the web to charging. Furthermore, some or all of the material layers may be treated with the antistatic material. In those instances where only some of the material layers are treated with antistatic material, the non-treated layer or layers may be subjected to charging prior to or after combining with the antistatic treated layer or layers.

To demonstrate the attributes of the present invention, the following Examples are provided.

Example 1

Kimberly-Clark manufactures a series of single sheet laminate nonwoven web materials made from three layers of fibrous material, i.e., spunbond-meltblown-spunbond (SMS) layers. These materials are available in a variety of basis weights. The two nonwoven webs used in these Examples were such single sheet laminate materials sold by Kimberly-Clark. Each of the nonwoven webs had a basis weight of 2.2 osy (ounces per square yard). Both spunbond layers had a basis weight of 0.85 osy and the meltblown layer had a basis weight of 0.50 osy. One of the nonwoven webs was a ZELEC® treated laminate and is sold by Kimberly-Clark the under the mark KIMGUARD® Heavy Duty Sterile Wrap and is designated in Table I as "KIMGUARD®".

The other nonwoven web, designated in Table I as "RSR" also had a basis weight of 2.2 osy but was not treated with an antistatic material. Both spunbond layers had a basis weight of 0.85 osy and the meltblown layer had a basis weight of 0.50 osy.

The method used to subject these webs to electrostatic charging (electret treating) is described in the above referenced U.S. Patent Application No 07/958,958.

The surface charge for both KIMGUARD® and RSR fabrics were analyzed and the data reported in Table I. The charge data for each side of these fabrics was recorded for both before ("AS RECEIVED") and after charging ("ELECTRETED"). Charge data were also recorded for ethylene oxide sterilized fabric samples which were first charged and then ethylene oxide sterilized ("AFTER EO TREATMENT"). As noted in Example 1, the KIMGUARD® samples were treated with ZELEC® and the RSR samples were not. Charge measurements were taken at 36 separate surface locations on each sample. For the categories, i.e., "AS RECEIVED" and "ELECTRETED", the KIMGUARD® and RSR samples were each single large sheets of material. Each such sheets were then portioned into several smaller samples. Sterilization and filtration data

reported in Example 2 were derived from these smaller samples.

Charge measurements reported are averaged values of positive (+) or negative (-) volts per cm^2 . The equipment
5 used to measure charge was an Electrostatic Voltmeter (Trek Model 344, Trek, Inc, Median, NY).

TABLE I

	<u>Material</u>	<u>Side</u>	<u>As Received</u>	<u>Electreted</u>	<u>After EO Treatment</u>		
					<u>Sample 1</u>	<u>Sample 2</u>	<u>Sample 3</u>
5							
10	KINGGUARD® (ZELEC®)	A	-2.8	-125	-4.2	27.2	-
		B	+1.6	- 15	24.1	-5.4	-
15							
	RSR (Non-ZELEC®)	A	- 61	+272	-89	-130	-138
20		B	- 87	-432	-90	- 46	+ 54

As illustrated by the above data, the ethylene oxide sterilization process generally diminished the overall surface charge for both the electret treated KIMGUARD and the RSR material.

5

Example 2

A summary of the average bacterial filtration efficiency (BFE) test results and standard deviation (SD) are reported for the two categories investigated for KIMGUARD® in Table II. The first category, reported in Table II is the "Nelson BFE". "Nelson BFE" stands for Nelson Laboratory's (Salt Lake City, UT.) bacterial filtration efficiency test. The procedure used to determine these BFEs is described in Nelson Laboratories' Protocol No. ARO/007B in accordance with MIL Spec 36954C, 4.4.1.1.1 and 4.4.1.2. This category includes the average BFE for 11 KIMGUARD® fabric samples which were electret-treated then ethylene oxide-sterilized ("KIMGUARD®/Electret/EO") and 11 non-electret-treated KIMGUARD® fabric samples which were ethylene oxide-sterilized ("KIMGUARD®/EO").

20

The second category reported in Table I is "Microbial Challenge BFE". This category includes the average BFEs for the KIMGUARD® samples.

The Microbial Challenge BFE procedure utilized a six port exposure chamber. Five of the ports accommodated five separate samples. The challenge control filter material was positioned in the sixth port. Three conditions were maintained in the microbial challenge test. These were: first, a 2.8 LPM (Liters Per Minute) flow rate through each of the ports; second, an exposure time of fifteen minutes followed by a chamber exhaust of fifteen minutes, and; third, a microbial challenge that results in 1×10^6 CFU's (Colony Forming Units) per port. *Bacillus subtilis* spores, purchased from Amsco (Part No. NA-026, P-764271-022) were used to make the working spore suspension of 1×10^6 CFUs per port recovery.

35

The value reported is an expression of the reduction of number of colony forming units (CFUs) or bacteria passing

through a sample compared to the number CFUs passing through the challenge control filter material. This value was derived by subtracting the number of CFUs passing through a sample from the number of CFUs passing through the challenge control filter material. The difference in the number of CFUs passing through these materials is then divided by the number of CFUs passing through the challenge filter material and then multiplied by 100 to convert to percent.

Table II

Sample	Nelson BFE	Microbial Challenge BFE
KINGGUARD®/Electret/EO	97.51+/-0.39	96.44+/-4.51
15 KINGGUARD®/EO	89.96+/-1.04	79.04+/-6.50

Table III summarizes the average Nelson BFE and the Microbial Challenge BFE categories for the RSR nonwoven materials. The procedures for both the Nelson BFE and Microbial Challenge BFE for the RSR materials were identical to the Nelson BFE and Microbial Challenge BFE procedures describe above. "RSR/Electret/EO" stands for RSR electret-treated then ethylene oxide-treated samples. "RSR/Electret" stands for RSR electret-treated samples. "RSR/EO" stands for RSR ethylene oxide-sterilized samples. 15 samples of each class of RSR material described above were analyzed and the results averaged.

TABLE III

Sample	Nelson BFE	Microbial Challenge BFE
30 RSR/Electret/EO	96.92+/-0.91	97.56+/-0.83
RSR/Electret	95.75+/-0.60	98.91+/-0.64
RSR/EO	79.73+/-3.20	79.82+/-5.96

35 Example 2 demonstrates that barrier properties of an ethylene oxide sterilizable material are improved when such material is first subjected to charging, and particularly

electrostatic charging, and then ethylene oxide sterilized as compared to the same material which is not subjected to charging prior to ethylene oxide sterilization. It will be further observed that the decrease in the surface charge
5 which occurred after ethylene oxide sterilization (Table I) did not significantly affect the barrier properties of these materials.

While the invention has been described in detail with respect to specific embodiments thereof, it will be
10 appreciated that those skilled in the art, upon attaining an understanding of the foregoing, may readily conceive of alterations to, variations of and equivalents to these embodiments. Accordingly, the scope of the present invention should be assessed as that of the appended claims
15 and any equivalents thereto.

What is claimed is:

1. A method of manufacturing a web comprising:
charging the web; and
ethylene oxide sterilizing the web.
2. The method of claim 1 wherein the web is a nonwoven web and is ethylene oxide sterilized prior to being charged.
3. The method of claim 2 wherein the nonwoven web is charged prior to being ethylene oxide sterilized.
4. The method of claim 2 wherein the charging is electrostatic charging.
5. The method of claim 1 further including the step of treating the web with an antistatic material.
6. The method of claim 1 wherein the web comprises first and second nonwoven webs joined together in juxtaposed relationship.
7. The method of claim 6 wherein the webs are joined after the charging step.
8. The method of claim 7 wherein the first web is charged and the second web is not charged.
9. The method of claim 6 wherein the first web is treated with an antistatic material.
10. The method of claim 9 wherein the second web is treated with an antistatic material.
11. An ethylene oxide sterilized web wherein portions of the web have been subjected to charging.

12. The web of claim 11 wherein the web is formed from a nonwoven material which comprises first and second nonwoven webs joined together in juxtaposed relationship.

13. The web of claim 11 having a surface and wherein such surface has a negative charge and wherein the average negative surface charge on the surface is less than 100 volts/cm².

14. The web of claim 11 having a first and a second surface and wherein these surfaces have a negative charge and wherein the average negative surface charge on the first surface is less than 100 volts/cm² and wherein the
5 average negative surface charge on the second surface is less than 100 volts/cm².

15. The web of claim 11 having a surface and wherein such surface has a positive charge and wherein the average positive surface charge on the surface is less than 60 volts/cm².

16. The web of claim 13 having an antistatic material present about portions thereof.

17. An ethylene oxide sterilized nonwoven web laminate comprising:

a two outer layers separated by an intermediate layer, wherein the two outer layers are spunbond nonwoven layers
5 and the intermediate layer is a meltblown layer; and
at least one of the layers is subjected to charging.

18. The nonwoven web of claim 17 wherein all three layers are subjected to charging.

19. The nonwoven web of claim 18 wherein at least one of the layers is treated with an antistatic material.

INTERNATIONAL SEARCH REPORT

International Application No

PC./US 95/07716

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 A61L2/20 B29C71/00 D04H13/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 A61L B29C D04H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP,A,0 391 725 (JOHNSON & JOHNSON) 10 October 1990 see claims 1-5 ---	1,4-7, 10,17
Y	US,A,4 761 326 (BARNES C.G.) 2 August 1988 see column 1, line 8 - line 15; claims 1-22 ---	1,4
Y	DATABASE WPI Week 8324 Derwent Publications Ltd., London, GB; AN 83-57499K & JP,A,58 076 118 (KOKEN KK) , 9 May 1983 see abstract --- -/--	1,5-7,9, 10

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

* Special categories of cited documents:

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Date of the actual completion of the international search

26 October 1995

Date of mailing of the international search report

15. 11. 95

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INTERNATIONAL SEARCH REPORT

International Application No

PC., US 95/07716

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US,A,4 863 785 (BERMAN M.H.S.) 5 September 1989 see column 2, line 5 - line 10 see column 5, line 11 - line 21 ---	17
Y	EP,A,0 550 029 (KIMBERLY-CLARK) 7 July 1993 see example 1 ---	9
A	WO,A,81 03265 (SURGIKOS) 26 November 1981 see page 1, line 12 - line 15; claims 1-9 -----	1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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